X-RAY OPTICS FOR TWO-DIMENSIONAL DIFFRACTION

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ABSTRACT

In two-dimensional X-ray diffraction the requirements of X-ray optics are different from the conventional diffractometer in many aspects. This paper discusses the performance requirements of various X-ray optics devices used for two-dimensional X-ray diffraction as well as commonly used X-ray optics components, such as monochromator, pinhole collimator, cross-coupled multilayer mirrors, UBC (universal beam concept) device, polycapillary and monocapillary.

INTRODUCTION

A two-dimensional X-ray diffraction (XRD$^2$) system has both the capability of acquiring diffraction patterns in 2D space simultaneously, and analyzing the 2D diffraction data accordingly [1-3]. In an XRD$^2$ system the function of X-ray optics is to condition the primary X-ray beam into the required wavelength, beam focus size, beam profile and divergency. Figure 1 shows a typical X-ray optics assembly for an XRD$^2$ system (GADDS – from Bruker AXS), which includes X-ray tube, monochromator, collimator and beamstop. It also shows the instrument center and the shadow of a fixed chi stage. Using a point X-ray source with pinhole collimation enables small samples or small regions on larger samples to be examined. A beamstop is placed behind the sample in transmission mode diffraction to prevent the direct beam from striking the detector. This configuration enables crystallographic phase, texture, and residual stress to be measured from precise locations on irregularly shaped parts, including curved surfaces.

Figure 1. Typical X-ray optics in a standard Bruker GADDS system.
This document was presented at the Denver X-ray Conference (DXC) on Applications of X-ray Analysis.

Sponsored by the International Centre for Diffraction Data (ICDD).

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The conventional diffraction, either using a point detector or a PSD, is confined within the diffractometer plane, the variation vertical to the diffractometer plan is not considered, the X-ray beam is normally extended in that direction (line focus). In XRD², a large portion of the diffraction rings are measured simultaneously. Since the diffraction patterns in all directions are equally important, the ideal X-ray beam profile is a circular spot (point focus). Most conventional diffractometers use the Bragg-Brentano parafocusing geometry, in which the sample surface normal is always a bisector between the incident beam and the diffracted beam. In an XRD² system, the diffracted X-rays are measured in a two-dimensional range so that the Bragg-Brentano geometry can not be achieved. In a conventional diffractometer, both the incident beam and diffracted X-rays can be conditioned before reaching the point detector. However, it is only possible to condition the incident beam for an XRD² system. Therefore, X-ray optics for XRD² systems has different requirements in terms of the beam spectrum purity, divergency and beam cross-section profile.

**X-RAY BEAM SHAPE FOR XRD²**

In principle, the cross-section shape of the X-ray beam used in an XRD² should be an infinitesimal point. In practice, the beam cross-section can be either round or square in limited size. If a line focus beam is used in a two-dimensional diffraction system, the smearing effect will dramatically increase the peak width, especially at the portion of diffraction ring away from the diffractometer plane. Figure 2a. is a diffraction frame of corundum collected with a line-focus incident beam. The diffraction rings are broadened at the portion away from diffractometer plane. As a comparison, Figure 2b shows the diffraction frame collected with a point beam, no smearing effect is observed. Therefore, all the diffraction rings can be used for data analysis.

![Figure 2.](image)

(a) smearing effect from line beam. (b) diffraction rings from point beam.
BEAM SPREAD OVER FLAT SAMPLE SURFACE (GEOMETRY BROADENING)

Conventional diffractometers use the Bragg-Brentano parafocusing geometry [4] as is show in Figure 3. A divergent beam from X-ray tube passes first through divergent slit, then hits the sample surface with an incident angle $\theta$. The incident X-rays spread over the sample surface with various incident angles in the vicinity of $\theta$. The area of irradiated region depends on the incident angle $\theta$ and beam divergence. The diffracted rays from the irradiated area leave the sample at an angle $2\theta$ from the corresponding incident rays, pass through the anti-scatter slit, and focus at detector slit. A point X-ray detector can be mounted at the position right after the detector slit or after a crystal monochromator. It can be seen that the beam-spread over the sample varies with the incident angle $\theta$, but the diffracted beam are focused back to the point detector as long as the sample surface normal bisect the incident and diffracted beams.

In an XRD$^2$ system, the diffracted X-rays are measured simultaneously in a two-dimensional range so that the Bragg-Brentano geometry can not be achieved. The beam-spread over the sample surface can not be focused back to the detector. Figure 4 shows the beam-spread in low incident angle over a flat sample surface observed by a two-dimensional detector. Figure 4a is in reflection mode, the diffracted beam in low $2\theta$ angle is narrower than the diffracted beam in high $2\theta$ angle. Figure 4b shows that the opposite is true in transmission mode.

![Figure 3. A conventional diffractometer in Bragg-Brentano geometry](image)

![Figure 4. Beam spread on a flat sample surface with a small incident angle. (a) reflection mode. (b) transmission mode.](image)
If the sample size is limited to the size comparable to the X-ray beam size, the peak broadening caused by beam spread can be reduced or eliminated. Loading powder sample in capillary is one way to achieve this effect. When collecting phase ID data with flat sample, set the sample $\omega$ angle as the half of medium $2\theta$ can also reduce the beam spread effect.

**AIR SCATTER AND FLUORESCENCE**

Air scatter in XRD$^2$ system has a significant contribution to the intensity background. In the conventional diffractometer, one can use anti-scatter slit (Figure 3), diffracted beam monochromator or soller slit to remove most air scatter not travelling in the diffracted beam direction. While all these measures can not be used for XRD2 system, which requires an open space between the sample and 2D detector. As is shown in Figure 5, air scatter is generated from the incident beam and diffracted beam. Obviously, the air scatter from the incident beam is significantly stronger than that from diffracted X-ray rays. The intensity of the air scatter from the incident beam is proportional to the length of the open incident beampath, which is the distance between the sample and the beam collimation exit. As is shown in Figure 1 for a typical optics design for GADDS, in order to reduce air scatter from the incident beam, the tip of the collimator is 6 mm from the sample. The air scatter from the diffracted X-rays is relatively weak, the intensity depends on sample-to-detector distance. It is typically not necessary to take measures to remove air scatter from the diffracted X-rays between the sample and 2D detector. However, if the sample-to-detector distance is large, for instance, 30 cm or above, it is necessary to use He-beampath or vacuum beampath to reduce air scatter.

![Figure 5. Air scatters from incident X-ray beam and diffracted X-rays.](image)

The radiation fluorescence is another source of intensity background in XRD$^2$, especially when the X-ray energy of the incident beam is slightly higher than the absorption edge of the sample elements. For example, when Cu-K$_\alpha$ radiation is used for iron or ferrous alloys. In a conventional diffractometer, fluorescence can be removed from the collected data by using a diffracted beam monochromator, or energy discrimination device. However, most two-dimensional detectors have a very limited energy resolution and it is impossible to add a diffracted beam monochromator in front of the 2D detector. The best way to avoid fluorescence is to choose a tube target having K$_\alpha$ energy lower then the absorption edge of the sample materials. For example, use Cr- K$_\alpha$ for ferrous alloys.
MONOCHROMATOR AND MULTILAYER MIRRORS

In a conventional diffraction system, the monochromator can be used either in the source side or the detector side, or both sides, while it is only possible to have a monochromator in the source side for an XRD$^2$ system. A crystal monochromator allows only a selected characteristic line to pass through, typically Kα radiation. Multilayer mirrors reflect X-rays in the same way as Bragg diffraction from crystals. Recent developments in X-ray optics include graded multilayer X-ray mirrors, known as Göbel Mirrors [5]. In contrast to conventional monochromator crystal, Göbel Mirrors are manufactured so that the d-spacing between the layers varies in a controlled manner. A crossed-coupled arrangement provides a highly parallel beam which is much more intense than can be obtained with a graphite monochromator. For applications such as microdiffraction where a small spot size is desired, Göbel Mirrors can offer up to an order of magnitude higher intensities than conventional optics. The low divergence of the beam incident on the sample from Göbel Mirrors also decreases the width of crystalline peaks and improves the resolution.

![Figure 6. Comparison of X-ray intensity between cross-coupled Göbel Mirrors and monochromator for various collimator sizes by experiment and simulation.](image)

Experimental results show that the smaller the beam size, the stronger the intensity gains from cross-coupled Göbel Mirrors compared with a monochromator (Figure 6). The intensity break-even point for Göbel Mirrors versus standard monochromator with pinhole collimation is approximately 0.3 to 0.4 mm. In other words, for applications, such as texture or phase identification from a bulk powdered specimen, which ordinarily employ collimators larger than 0.4 mm, there is no benefit to using Göbel Mirrors. In fact, the low divergence of the resulting beam can cause poor statistical grain sampling in such cases. Therefore, the cross-coupled Göbel Mirrors are especially suitable for microdiffraction and small angle X-ray scattering. The universal beam concept (UBC) device, developed by Bruker AXS, use a single multilayer mirror coupled with line focus X-ray source, which can be easily switched between line focus and point focus by using slit or collimator.
PINHOLE COLLIMATOR AND MONOCAPILLARY

The pinhole collimator is used to control the beam size and divergence. In an XRD$^2$ system, the pinhole collimator is normally used together with a monochromator or a set of cross-coupled Göbel mirrors. The beam divergency decreases continuously with decreasing pinhole size for the combination of double pinhole collimator and monochromator [6]. For quantitative analysis, texture, or percent crystallinity measurements, 0.5 mm or 0.8 mm collimators are typically used. In the case of quantitative analysis and texture measurements, using too small a collimator can actually be a detriment, causing poor statistical grain sampling. In such cases, statistics can be improved by oscillating the sample.

Capillary X-ray optics is based on the concept of total external reflection. X-rays can be reflected by a smooth surface when the angle of incidence is smaller than the critical angle for total reflection $\theta_c$. The critical angle is a function of the wavelength and materials; the shorter the wavelength, the lower the critical angle. When X-rays are reflected by the inner surface of a capillary at a grazing angle smaller than the critical angle of the capillary materials, X-rays are reflected with little energy loss. It can produce significant intensity gain on the sample relative to the pinhole collimators. The following table list the Intensity gain (calculated and experimental) and beam spot size including 90% energy on sample for monocapillaries compared with double pinhole collimators

<table>
<thead>
<tr>
<th>Capillary/Pinhole size: $d$ (mm)</th>
<th>Cu-K$_\alpha$-radiation (8.0keV)</th>
<th>Mo-K$_\alpha$-radiation (17.4keV)</th>
<th>Collimator</th>
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<tbody>
<tr>
<td></td>
<td>Gain-cal</td>
<td>Gain-exp</td>
<td>Spot(90%)</td>
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REFERENCES